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Influence of Nitrogen on the Notch Toughness of Heat-Treated 0.3-Percent-Carbon Steels at Low Temperatures[1]

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Charpy impact tests were made at temperatures ranging from −196° to +100° C on fully hardened and tempered specimens of 0.3-percent-carbon steels with variable nitrogen. The transition from ductile to brittle fractures was affected by both the amount and form of the nitrogen in the steels. Fixation of the nitrogen as aluminum nitride was beneficial, as the aluminum-treated steels had considerably lower transition temperatures than those of the steels not treated with aluminum.

1. Introduction

It is now widely recognized in the field of metallurgy that ferrous metals, such as ordinary carbon and low-alloy steels, exhibit a characteristic loss of toughness when certain low temperatures are reached. This decrease in the toughness of the steel may occur near room temperature or at much lower temperatures, depending upon the composition, manufacturing process, subsequent treatment of the steel, and method of applying stress. Under the conditions of stress imposed by a notch and at some temperatures or range of temperatures, the behavior of the steel can be expected to change from tough to brittle. This change is generally accompanied not only by a decrease in ductility but also by a change in the appearance of the facture surface of the steel, the fracture surface changing from a fibrous to a granular type as the temperature is lowered from above to below this transition.

Different methods of testing are employed for determining the transition temperature of various materials [1 to 8].[2] A convenient and widely used method consists of breaking notched specimens, such as Charpy V- or keyhole-notch, in impact at accurately controlled temperatures. However, it should be pointed out that the transition temperature as determined by one method may not agree closely with that obtained with some other method. In general, the transition temperature is lowered as the test conditions are changed from fracturing in impact to tension, and still lower values are obtained in torsion. As the primary purpose of the present investigation was to study the specific influence of nitrogen on the notch toughness at low temperature of fully hardened and tempered specimens of 0.30-percent-carbon steels, a single test method was used. This eliminated the many variable factors arising from differences in test methods. The results to be reported are those obtained on Charpy V-notch specimens fractured at temperatures ranging from −196° to +100° C.

2. Previous Investigations

Many investigators have reported studies on the notch toughness of ferritic steels in which the effects of oxygen, nitrogen, and aluminum have been discussed. Results of several investigations [3, 9 to 12] indicate that oxygen in the steel in solution or as iron oxide, manganese oxide, etc., has a strongly detrimental effect on the mechanical properties at low temperatures. Deoxidation of steel with aluminum, silicon, vanadium, titanium, or zirconium generally improves the mechanical properties at low temperatures [9, 11, 13 to 21]. As most of these deoxidizers are also effective as grain refiners and several also tend to combine with the carbon and nitrogen to form carbides and nitrides, their influence on the notch toughness of steel cannot be attributed solely to the removal of the oxygen. The notch toughness increases with decrease in the ferrite grain size [22 to 26], and it also may be affected by the amount and form of the nitrogen in the steel.

Fast [9] found that the energy absorbed by high-purity iron in notched-bar impact tests at room temperature decreased with increase in the amount of oxygen present; the effect was small for iron in which the carbon is 0.002 percent or more; the addition of 0.018 percent of oxygen to the iron raised the transition temperature about 50 deg C. Allen [12] reported from the results of tests made with Charpy V-notch specimens, that the transition temperature of normalized high-purity iron was affected markedly by its oxygen content; as soon as the oxygen increased above 0.003 percent, the transition temperature began to rise sharply. This notch brittleness in impact is attributed by both Fast and Allen to a grain boundary film of iron oxide, causing a weakening of the cohesion at the grain boundaries.

According to Fast [9] the presence of nitrogen in high-purity iron does not appreciably affect the low-temperature properties, but it is the cause of blue brittleness, that is, a decrease in absorbed energy in notch-bar impact tests in the range 360° to 460° C. Other investigators [3, 10, 27] also have attributed the blue brittleness of steel to nitrogen or nitrogen precipitates. However, Hultgren and Chang [28] recently reported that an iron nitride precipitate is not a cause of embrittlement of steel. The data obtained in many investigations [11, 15, 27, 29 to 34] indicate that nitrogen increases the aging and strain sensitivity of certain steels and irons. Some of these data also indicate that nitrogen decreases the notch-bar impact values and increases the transition temperatures. Nitro-

[1] This investigation was sponsored in part by the Ordnance Department, Watertown Arsenal.

[2] Figures in brackets indicate the literature references at the end of this paper.

gen influences the grain size of steel, and this affects the low-temperature properties. Sufficient data are not yet available to permit a separate analysis of these effects.

The beneficial action of aluminum on the toughness of steels at low temperatures has been attributed by several investigators [11, 15, 29, 30] to the fixation of the nitrogen as aluminum nitride. Nitrogen in the form of iron nitride or manganese nitride is believed to contribute more to strain sensitivity and embrittlement than nitrogen present as aluminum nitride. Work and Enzian [11] postulated that the presence of aluminum nitride in steel may even have a beneficial effect. They stated, "It seems probable that the form in which oxygen or nitrogen is present in the steel has a greater bearing on the properties than the total content of either element."

3. Steels and Procedures

The composition of the steels used is given in table 1. Each steel contained about 0.30 percent of carbon, 0.25 percent of silicon, and about 0.9 or 1.6 percent of manganese. The nitrogen and oxygen contents varied with the practice used in processing the heats.

The procedure for processing these steels is described in some detail in previous reports [35, 36]. Essentially, the procedure consisted of using ingot iron as the base of 200- to 300-lb charges, melting in a magnesia-lined induction furnace without slags, and pouring into big-end-up steel molds of about 50-lb capacity each, equipped with hot tops; steel 41 was prepared from a melt under slag. In preparing the series varying in nitrogen content, additions of calcium cyanamide (technical grade) were made to the molten charge just before deoxidizing with 0.10 percent of aluminum and pouring. Each ingot was hot-rolled into a plate 0.5 to 0.6 in. thick, and all test specimens were prepared from these plates after normalizing (heating at 1,650° F for 1 hr, followed by cooling in air). Metallographic examination of the normalized plates showed moderate to pronounced ferrite banding.

The Charpy specimens were rough machined to approximately 0.43 in. square by 2.16 in. long. These over-sized specimens were placed in a furnace at 1,575° F, and, after 30 min, were quenched in water at room temperature, tempered at 1,000° F for 1 hr, and air-cooled to room temperature. At 1,575° F, the steels treated with aluminum were fine-grained, whereas the steels not treated with aluminum were of mixed grain size (table 2). Because of this variation in grain size at 1,575° F, additional specimens of the steels not treated with aluminum were quenched from a lower temperature (1,500° F) at which they were also fine-grained. The heat-treated specimens were then wet ground to size (ends not ground) and notched with the notch located at right angles to the rolled surfaces of the plates and normal to the direction of rolling, as illustrated in figure 1.

Hardness tests (Rockwell C) were made at room temperature on the Charpy specimens after fracturing; two or more readings were made on each specimen. Each hardness value given in table 2 is the average obtained from the entire number of specimens of that steel. The hardness was quite uniform in all the specimens heat-treated from a selected steel, but the average hardness of the 0.9-percent-manganese steels was slightly lower than

TABLE 1. *Chemical composition of steels—percentage by weight*

The ingots and determinations for carbon, manganese, phosphorus, sulfur, and silicon were made at the Battelle Memorial Institute. These elements were determined on drillings from the ingot at the base of the hot top. Analyses for nitrogen, oxygen, hydrogen, aluminum, aluminum nitride, and aluminum oxide were made at the National Bureau of Standards on samples prepared from the fractured Charpy specimens by methods as follows: Total nitrogen, oxygen, and hydrogen by vacuum fusion; aluminum nitride as described by Beeghly [37]; aluminum oxide by solution in HNO_3; aluminum by spectrochemical analysis. The determinations for the gases and their compounds are considered to be accurate to within approximately ±0.001 percent.

Steel	Carbon	Manganese	Phosphorus	Sulfur	Silicon	Aluminum				Oxygen		Nitrogen		Hydrogen	Al_2O_3	AlN
						Total	As Al_2O_3	As AlN	Uncombined [a]	Total	As Al_2O_3	Total	As AlN			
Steels not treated with aluminum																
33	0.28	1.59	0.011	0.034	0.25	0.006	>0.001	0.002	>0.004	0.011	>0.001	0.006	0.001	0.0001	>0.001	0.003
37	.30	1.61	.008	.030	.25	.005	>.001	.004	>.001	.009	>.001	.005	.002	.0002	.001	.006
110	.29	1.64	.008	.029	.30	.004	>.001	.002	>.003	.011	>.001	.007	.001	>.0001	.001	.003
Steel treated with 0.05% of aluminum																
78	0.30	1.64	0.011	0.027	0.24	0.034	0.006	0.014	0.014	0.008	0.006	0.007	0.007	0.0001	0.011	0.021
Steels treated with 0.1% of aluminum																
41	0.30	1.66	0.010	0.020	0.20	0.056	0.009	0.008	0.042	0.008	0.008	0.004	0.004	0.0001	0.016	0.012
138	.30	1.68	.010	.029	.29	.068	.006	.014	.048	.006	.006	.008	.007	.0002	.012	.021
144	.30	1.56	.009	.030	.22	.056	.005	.015	.036	.004	.004	.008	.008	>.0001	.009	.023
160	.32	1.62	.011	.028	.23	.067	.005	.031	.031	.005	.005	.026	.016	>.0001	.010	.047
180	.29	1.64	.012	.026	.21	.076	.011	.046	.019	.010	.009	.027	.024	>.0001	.020	.070
17	.29	.92	.010	.021	.24	.051	.005	.010	.036	.004	.005	.004	.005	>.0001	.010	.015
189	.32	.92	.010	.026	.25	.064	.009	.029	.026	.007	.006	.016	.015	>.0001	.017	.044
198	.31	.92	.013	.031	.33	.074	.009	.060	.006	.007	.006	.032	.031	>.0001	.017	.091

[a] Total aluminum minus aluminum combined as oxide and nitride.

that of the steels containing 1.6 percent of manganese.

The notched-bar impact tests were carried out in duplicate at temperatures ranging from −196° to +100° C in a Charpy machine of 224.1 ft-lb capacity, with a striking velocity of the hammer of 16.85 ft/sec. The specimens, except those tested at room temperature, were immersed in an insulated bath at the desired temperature for a minimum time of 30 min and then quickly transferred to the impact machine and broken. The total time elapsing between the removal from the bath and the breaking of the specimen in the impact machine ranged from 3 to 4 sec. For the tests at −196° C, the insulated bath contained liquid nitrogen. For the tests at −120° and −100° C, the insulated bath contained dichloro-difluoro methane (Freon 12), and the desired temperature was maintained by the controlled passage of liquid nitrogen through a copper coil immersed in the bath. For the tests at −78°, −70°, −40°, and −35° C, the insulated bath contained equal parts by volume of carbon tetrachloride and chloroform, and the desired temperature was maintained by regulated additions of solid carbon dioxide. The temperatures of the specimens in the refrigerant bath were measured by means of a thermocouple and a precision potentiometer. For the tests at +100° C, the specimens were heated in a bath of boiling water in which a small amount of sodium chromate had been added as an inhibitor to corrosion.

4. Results and Discussion

The relation between the energy absorbed in fracturing Charpy V-notch specimens and test temperature for the different steels is shown in figures 2 to 5. In these figures, the value for energy re-

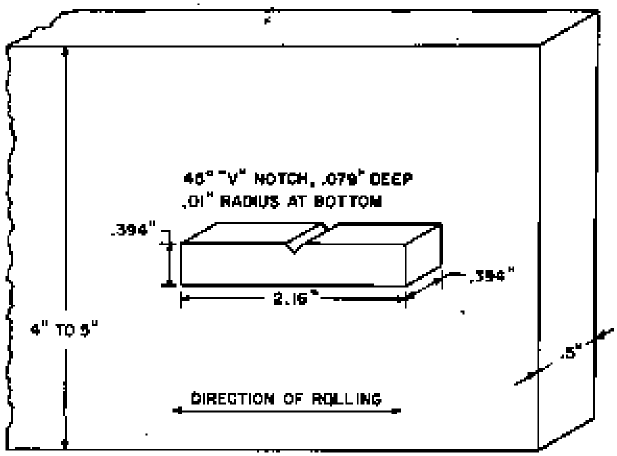

Figure 1. *Dimensions of Charpy specimen and its location in relation to the direction of rolling of the plates.*

quired to fracture each specimen is plotted against the test temperature, and smooth curves are constructed to represent the energy-temperature relationship. An interesting feature is the close agreement of the notch toughness values for duplicate tests even in the transition temperature range in which considerable scatter is normally expected.

Various criteria have been used for evaluating the data obtained in notch-bar impact tests, and no single criterion or group of criteria has been accepted as a standard. The specific criterion used depends primarily upon the type of test and objective of the investigation. In the present study, the evaluation of the test data is based mainly on the curve showing the relation between energy absorbed and test temperature, as given in figures 2 to 5. The transition

Table 2. *Heat treatment, austenite grain size, hardness, transition temperature, and energy absorbed at 20° C of Charpy V-notch specimens*

Steel	Type	ASTM grain No.[a]	Hardness, Rockwell C	Transition temperature		Energy absorbed at 20° C
				Energy absorbed [b]	Fracture appearance [c]	
Heat treatment A; quenched from 1,575° F, tempered at 1,000° F						
				° C	*° C*	*ft-lb*
33	1.6%-Mn steel, not treated with Al	4 (50%) 6–7 (50%)	27	−44	−44	44
37	do	4 (50%) 7 (50%)	28	−49	−43	42
110	do	4–5 (50%) 6–7 (50%)	28	−44	−38	42
76	1.6%-Mn steel, treated with 0.05% Al	8	27	−85	−88	50
41	1.6%-Mn steel, treated with 0.1% Al	8	27	−80	−80	65
138	do	8	27	−81	−88	54
144	do	8	25	−83	−86	59
150	do	8	28	−90	−90	50
180	do	8	27	−92	−92	45
17	0.9%-Mn steel, treated with 0.1% Al	8	24	−84	−87	69
168	do	8	25	−82	−87	61
196	do	8	26	−96	−85	49
Heat treatment B; quenched from 1,500° F, tempered at 1,000° F						
33	1.6%-Mn steel, not treated with Al	7–8 (few 4–6)	27	−44	−44	44
37	do	7 (few 6)	28	−53	−42	42
110	do	7–8 (few 6)	28	−44	−38	42

[a] At austenitizing temperature.
[b] Based on the mean value of energy absorbed at +100° and −196° C.
[c] Based on 50% area of granular fracture.

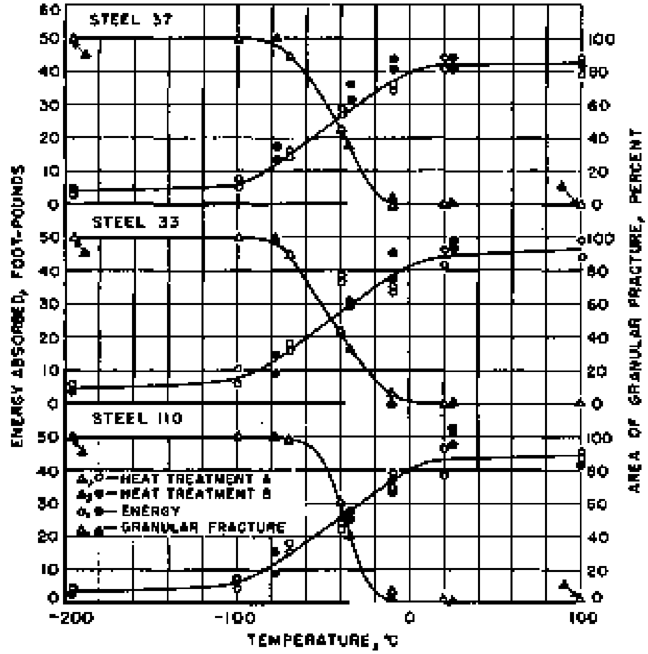

Figure 2. *Relations of testing temperature to the energy absorbed and appearance of the fracture of Charpy V-notch specimens of 0.3-percent-carbon, 1.6-percent-manganese steels, not treated with aluminum.*

Refer to table 2 for heat treatments A and B.

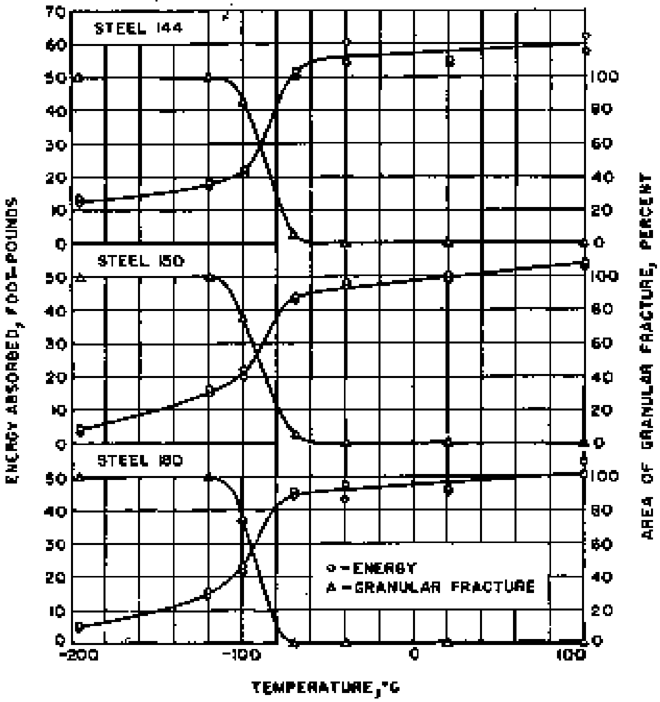

Figure 4. *Relations of testing temperature to the energy absorbed and appearance of the fracture of Charpy V-notch specimens of 0.3-percent-carbon, 1.6-percent-manganese steels, treated with aluminum.*

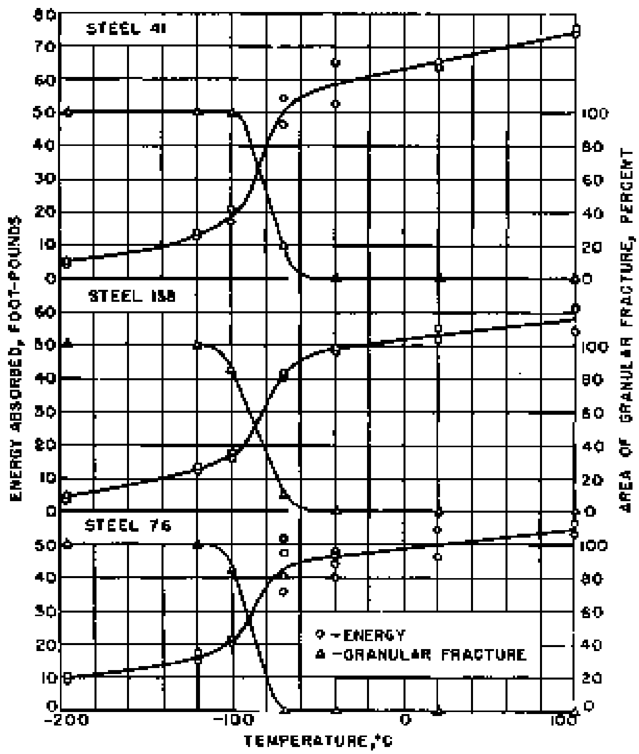

Figure 3. *Relations of testing temperature to the energy absorbed and appearance of the fracture of Charpy V-notch specimens of 0.3-percent-carbon, 1.6-percent-manganese steels treated with aluminum.*

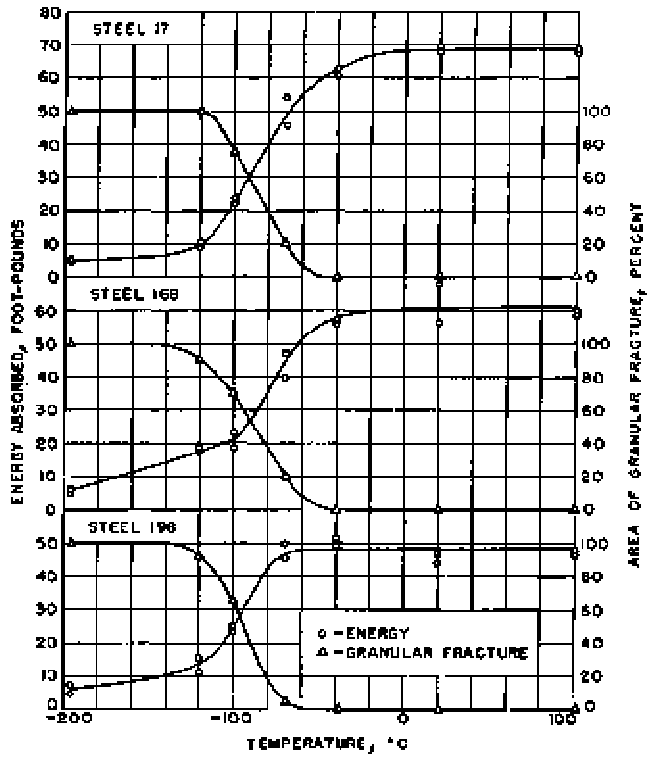

Figure 5. *Relations of testing temperature to the energy absorbed and appearance of the fracture of Charpy V-notch specimens of 0.3-percent-carbon, 0.9-percent-manganese steels, treated with aluminum.*

temperature from tough to brittle behavior is designated as the temperature at which the energy absorbed is the mean of the highest and lowest values, that is, mean value for tests made at +100° and −196° C, respectively. The mean energy value occurs approximately at the position of the greatest slope of the curve. Thus this transition temperature also corresponds to that at which the rate of decrease in energy is a maximum with decrease in test temperature.

The appearance of the fractured surfaces of Charpy V-notch specimens also shows a correlation with the shape of the above curves. A dull, fibrous appearance with considerable deformation is characteristic of Charpy impact fractures of a tough material, whereas a bright granular or crystalline appearance with very small deformation is typical of a brittle material. Partly fibrous and partly granular fractures are often obtained in specimens tested in the transition range from tough to brittle material. This correlation is evident by a comparison of the curve representing the estimated percentage of granular fracture at each test temperature (average of duplicate specimens) with the curves based on energy values. A transition temperature of each steel when based upon 50-percent granular fracture corresponds fairly closely to that as determined by the energy-temperature curve (table 2).

The influence of variation in the chemical composition of the 0.3-percent-carbon steels used in this investigation on the transition temperature and the notch toughness at low temperatures is summarized in figures 6 to 12. The straight lines shown in these figures were determined by the method of least squares.

The influence of nitrogen on the notch toughness at room temperature and on the transition temperature of the aluminum-treated steels is shown by the data given in figures 6 and 7. The slope of the straight line for the notch toughness at room temperature of the 0.9-percent-manganese steels (fig. 6) is significantly different from zero,[2] whereas the slope of the line for the 1.6-percent-manganese steels is not significantly different from zero. Although there was a slight trend with the 0.9-percent manganese steels for the energy absorbed at room temperature to decrease as the nitrogen was increased, this trend is not believed to be of practical importance; all of these aluminum-treated steels are classified as notch tough at room temperature (range of about 50 to 70 ft-lb). However, a definite and significant trend is shown in figure 7 in that the transition temperature of these steels was lowered as the nitrogen was increased.

Both iron and manganese nitrides have been reported as being detrimental to notch toughness at low temperature. Chemical analyses, therefore, were made to ascertain the form of the nitrogen in the present steels. The results given in table 1 show that the nitrogen in the aluminum-treated steels was in the form of aluminum nitride. Thus, when the aluminum nitride is plotted against the energy absorbed at room temperature and the transition temperature, relationships are obtained that are similar to the above, as is illustrated for the latter in figure 8. This evidence, therefore, indicates that high nitrogen present in the form of aluminum nitride was beneficial to the notch toughness at low temperature of these 0.3-percent-carbon steels, containing either 0.9 or 1.6 percent of manganese. Sufficient data are not available to determine the role of dissolved (uncombined) nitrogen on notch toughness of these steels.

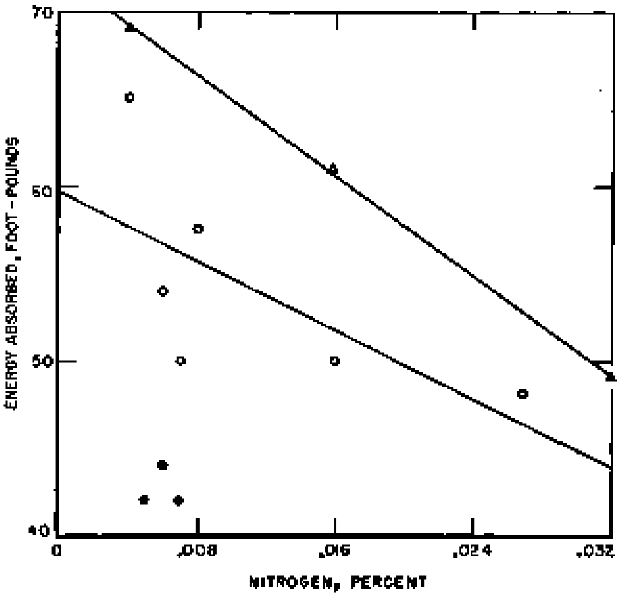

FIGURE 6. *Effect of the total nitrogen content of 0.3-percent-carbon steel on the energy absorbed in impact tests at room temperature with Charpy V-notch specimens.*

●, 1.6-percent-manganese steels, not treated with aluminum; ○, 1.6-percent-manganese steels, treated with aluminum; △, 0.9-percent-manganese steels, treated with aluminum.

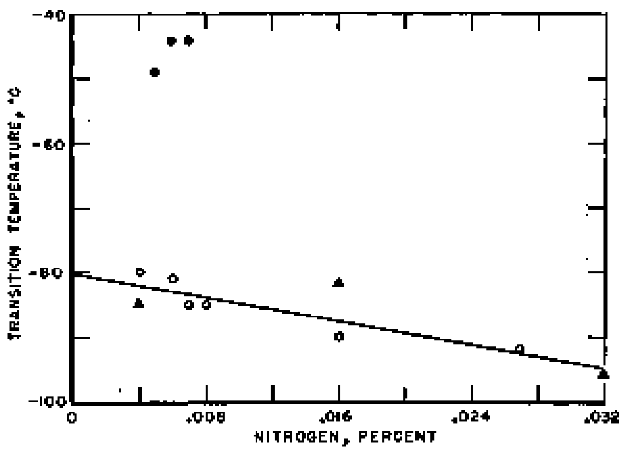

FIGURE 7. *Effect of the total nitrogen content of 0.3-percent-carbon steel on the transition temperature of Charpy V-notch specimens.*

●, 1.6-percent-manganese steels, not treated with aluminum; ○, 1.6-percent-manganese steels, treated with aluminum; △, 0.9-percent-manganese steels treated with aluminum.

[2] The method used to determine significance is essentially that reported by Youden [38].

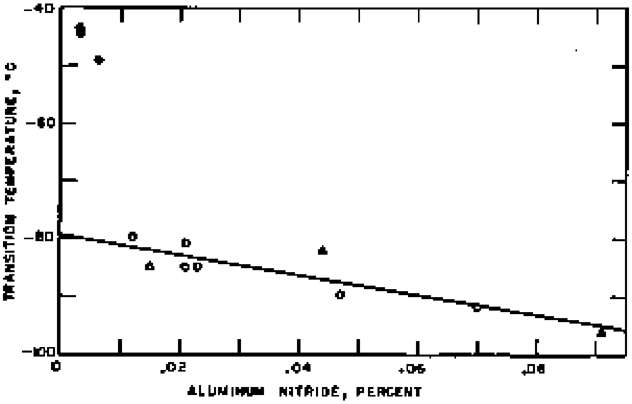

FIGURE 8. *Effect of the aluminum nitride content of 0.8-percent-carbon steel on the transition temperature of Charpy V-notch specimens.*

●, 1.6-percent-manganese steels, not treated with aluminum; ○, 1.6-percent-manganese steels, treated with aluminum; △, 0.9-percent-manganese steels, treated with aluminum.

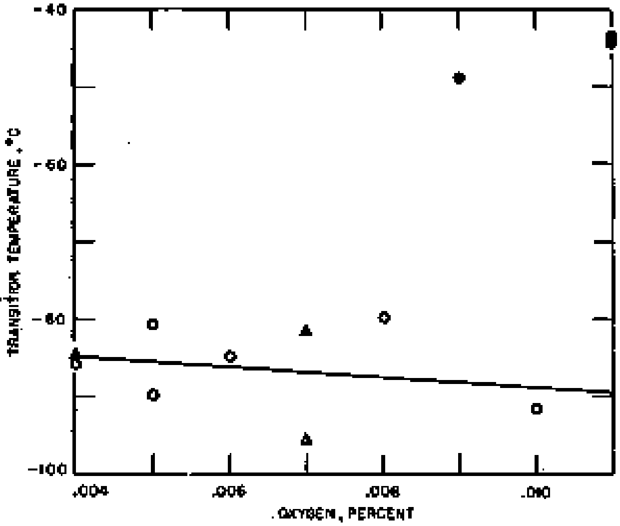

FIGURE 9. *Effect of the total oxygen content of 0.8-percent-carbon steel on the transition temperature of Charpy V-notch specimens.*

●, 1.6-percent-manganese steels, not treated with aluminum; ○, 1.6-percent-manganese steels, treated with aluminum; △, 0.9-percent-manganese steels, treated with aluminum.

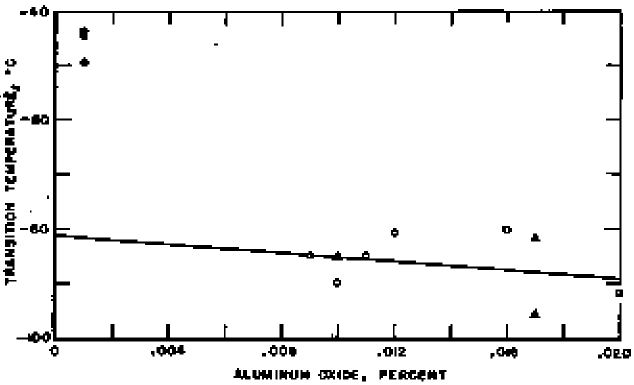

FIGURE 10. *Effect of the aluminum oxide content of 0.8-percent-carbon steel on the transition temperature of Charpy V-notch specimens.*

●, 1.6-percent-manganese steels, not treated with aluminum; ○, 1.6-percent-manganese steels, treated with aluminum; △, 0.9-percent-manganese steels, treated with aluminum.

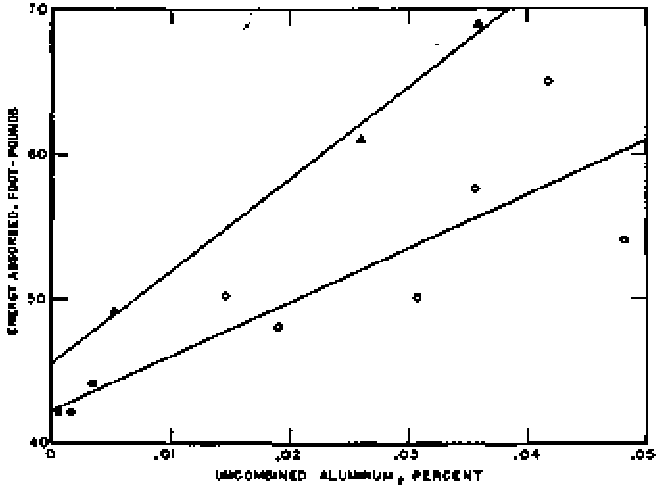

FIGURE 11. *Effect of the uncombined aluminum content of 0.8-percent-carbon steel on the energy absorbed in impact tests at room temperature with Charpy V-notch specimens.*

●, 1.6-percent-manganese steels, not treated with aluminum; ○, 1.6-percent-manganese steels, treated with aluminum; △, 0.9-percent-manganese steels, treated with aluminum.

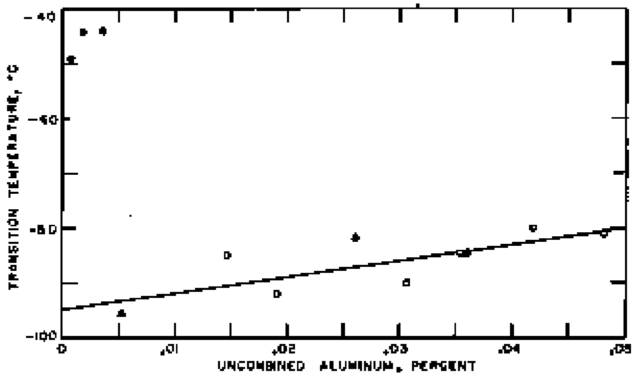

FIGURE 12. *Effect of the uncombined aluminum content of 0.8-percent-carbon steel on the transition temperature of Charpy V-notch specimens.*

●, 1.6-percent-manganese steels, not treated with aluminum; ○, 1.6-percent-manganese steels, treated with aluminum; △, 0.9-percent-manganese steels, treated with aluminum.

A noteworthy feature is the relatively high transition temperature of the three steels not treated with aluminum (values shown as dark circles in figs. 6 to 12). As the notch toughness of certain heat-treated steels is known to be affected by wide variations in austenite grain size, and as the average grain size at 1,575° F of the steels not treated with aluminum was coarser than that of the aluminum-treated steels (table 2), additional tests were made on fine-grained Charpy specimens of the former steels when quenched from 1,500° F, followed by tempering at 1,000° F. The results, summarized in figure 2 (compare results of heat treatments A with B) show only minor, if any, decreases in transition temperature with a change in austenite grains from ASTM No. 4–5 to No. 7–8. The observed difference in transition temperatures between the steels treated with aluminum and the steels not treated with aluminum, therefore, cannot be attributed to a grain size effect.

Metallographic examination of the cross sections prepared from each of the heat-treated steels (all steels included in table 1) showed no indication of free ferrite. Evidently, the relatively high transition temperature of the steels not treated with aluminum is not due to the presence of free ferrite in the heat-treated Charpy specimens.

It is believed that the inferior properties at low temperatures of the three steels not treated with aluminum may be attributed to the presence of nitrides and oxides other than aluminum, or a combination of these compounds.

The total oxygen content of the steels treated with aluminum ranged from about 0.004 to 0.010 percent. This oxygen existed in the heat-treated specimen principally as aluminum oxide (table 1). In the steels not treated with aluminum, the total oxygen ranged from 0.009 to 0.011 percent, with less than 0.001 percent as aluminum oxide. For the steels treated with aluminum, an apparent trend was indicated for the transition temperature to decrease slightly with an increase in total oxygen (fig. 9) or aluminum oxide (fig. 10). However, the slope of each straight line is not significantly different from zero, and thus for these steels variation in either total oxygen or aluminum oxide had no appreciable effect on the transition temperature. Moreover, variation in either total oxygen or aluminum oxide had no significant effect on the notch toughness at room temperature of these steels.

No significant trends were found when the total aluminum was plotted against the transition temperature or the energy absorbed at room temperature. Trends were shown for both the energy absorbed at room temperature and the transition temperature to increase as the amount of uncombined aluminum was increased, as is illustrated in figures 11 and 12, respectively. The slope of each line is significantly different from zero. The data indicate that the presence of aluminum in excess of that necessary to fix all of the oxygen and nitrogen in these 0.3-percent-carbon steels as aluminum oxide and aluminum nitride was slightly beneficial to notch toughness at room temperatures and detrimental at low temperatures. However, it should again be pointed out that these steels contained variable amounts of nitrogen in the form of aluminum nitride and, to a rough approximation, the aluminum nitride decreased with increase in the uncombined aluminum content. Thus, these trends also can be attributed to the variation in the aluminum-nitride content of the steels. Moreover, it should be noted that the transition temperature of the steels not treated with aluminum were considerably higher than those of the steels treated with aluminum. The inferior properties of the steels not treated with aluminum presumably might be due to the presence of nitrides of iron or manganese or both.

Increasing the manganese from 0.9 to 1.6 percent had no material effect on the transition temperature of this series of steels with variable nitrogen. The energy absorbed at room temperature was slightly higher in the 0.9-percent-manganese than that of the 1.6-percent-manganese steels with the same total nitrogen (fig. 6) or uncombined aluminum (fig. 11). As previously pointed out, however, the average hardness of the Charpy specimens of the the 0.9-percent-manganese steel was slightly lower than that of the 1.6-percent-manganese steel.

5. Summary

Charpy impact tests were made at temperatures ranging from −196° to +100° C on fully hardened and tempered specimens (V-notch) of 0.3-percent carbon, 0.9- and 1.6-percent manganese steels with variable nitrogen. All steels were made in induction furnaces.

The steels that had been treated with 0.10-percent aluminum in the furnace just prior to pouring had better notch toughness at low temperatures, as measured by the transition temperature at which the fracture changes from ductile to brittle, than the steels not treated with aluminum. The notch toughness at room temperature of the aluminum-treated steels also was somewhat superior to that of the steels not treated with aluminum.

The evidence indicates that the influence of nitrogen on notch toughness at low temperatures was affected not only by the amount present but also by its form. A trend was observed of a lowering of the transition temperature with increase in the aluminum nitride content of the steels treated with aluminum in excess of that necessary to fix all of the nitrogen and oxygen as aluminum nitride and aluminum oxide, respectively. The relatively high transition temperature of the steels not treated with aluminum may be attributed to nitrides other than aluminum, oxides other than aluminum, or a combination of these compounds.

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